QUALITY ASSURANCE PROJECT PLAN FOR AIR TOXIC SAMPLING IN AND AROUND THE ALTEN ETHANOL FACILITY, MEAD, NE

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A1. Title & Approval Sheet

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A.2 TABLE OF CONTENTS

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A.4 Project/Task Organization

This document has been prepared to describe the procedures to be used by the EPA Region 7, Laboratory Services and Applied Science Division (LSASD), Field Service Branch (FSB) personnel to conduct air toxics sampling in and around the AltEn Ethanol Facility, Mead, NE in cooperation with the Nebraska Department of Environment and Energy (NDEE) personnel. Thermal Desorption (TD) Tubes and Sorbent tubes (XAD-2) will be utilized to collect several air samples which will be sent to the US EPA Officer of Research and Development (ORD) for laboratory analysis to potentially characterize unidentified odors and air toxics.

A.5 Problem Definition / Background

The purpose of this Quality Assurance Project Plan (QAPP) is to describe the procedures to be used for the air sampling event to screen potential unidentified existing air toxics at the AltEn Ethanol facility located in Mead, NE. This air sampling will focus on identification of the contaminants causing strong odors in and around the facility. This study was initiated at the request of the Nebraska Department of Environment and Energy (NDEE).

The AltEn ethanol plant opened in January 2015 in Mead, NE, a farming community located 40 miles west of Omaha. The company focused on a process producing ethanol from the surplus corn seed that seed companies produce on an annual basis. As a result, AltEn became one of the largest stockpilers of surplus seed corn in the United States. This surplus seed corn was coated with fungicides and insecticides, including those known as neonicotinoids. The ethanol production process, when applied to treated seed, creates waste laced with chemicals that researchers say may be harmful to people, animals, and the environment. The current two primary concerns are distiller's grain (wetcake) and the wastewater lagoons that are located on the AltEn facility site. In 2019, the wetcake (sold to farmers as fertilizer) was found to be contaminated with levels of toxins in excess of 1,000 times the legal threshold.

EPA Region 7 and NDEE conducted air sampling at several sites in and around the AltEn Ethanol facility at Mead, NE on September 16-17, 2021, utilizing pressurized summa cannisters. The results did not indicate any TO-17 levels of concern.

NDEE is the lead regulatory agency over the AltEn facility and has conducted previous monitoring, including water, air, waste material, and soil sampling. The state has authorized several seed companies to develop a Remedial Action Plan under the NDEE Voluntary Cleanup Program.

A.6 Project / Task Description

The objective of the sampling project is to utilize TD tubes and XAD sorbent tubes to perform air sampling around the AltEn ethanol facility (Mead, NE) in the Summer 2022 as a screening tool to identify the sources of the existing odors being emitted. The results will provide a follow-up to the R7 EPA September 2021 Summa Cannister air sampling and R5 EPA GeoMap results.

A.6.1 Thermal Desorption (TD) Tube/XAD Sorbent Tube Methodology

Air samples will be conducted for unidentified odors by using an air sampling pump (Gillian plus) and TD/XAD sorbent tube technology. Each pump will be paired with a TD/XAD sorbent tube and pre-set with a specific flow rate of 200 cc/minute for each respective sample for a duration of 30 minutes. At the time of deployment at each sampling location, each pump will be flow calibrated using a MesaLabs Defender 530. Flow calibration, flow readings, and start times for each TB/sorbent tube sampling set up will be recorded onto a sampling worksheet (Attachment A). A description of each type of sampling tube and process is as follows:

Thermal Desorption (TD) Tubes - High-quality thermal desorption tubes are suitable to sample for ppt to ppm concentrations of volatile organic compounds (VOCs) in ambient air. To use, the sampling tube is connected to a small vacuum pump that draws air through the tube at a precise flow rate over a specific period of time. Thermal desorption tubes are packed with adsorbent materials that trap VOCs. Specific air sampling methodology for the use of TD Tubes on this project can be found in the "Compendium of Methods for the Determination of Toxic Organic Compounds in Ambient Air Second Edition, Compendium Method TO-17" (EPA/625/R-96/010b).

The standard operating procedure for this method's sample collection is summarized as follows:

- Record the current sampling location on the appropriate field form (include GPS coordinates).
- Calibrate each personal sampling pump with a representative TD sample tube in line, record flow rate.
- Take off the SwedgeLock brass caps from each end of the TD sampling tube immediately prior to sampling. Attach sampling tube to pump with flexible tubing.
- Sample at an accurately known rate for a specific duration.

- Place the SwedgeLock brass caps back on each end of the sampling tube and pack securely for shipment with bagged refrigerant. Do not shut down the pump at this time. Each tube should go in a labeled Ziploc bag, noting sample ID and parameter, date and time. Labels should be prepared in such a way that they will not be ruined by water or refrigerant. Samples may be stored at 4C until they are shipped.
- Record the sampling information onto the Chain of Custody form.
- Using the same setup as for initial calibration, measure the flow rate again with the same dedicated TD tube specific to the pump used for that method. Record the final flow rate onto the Air Sampling worksheet.

XAD Sorbent Tubes – XAD Sorbent tubes are used to measure the average concentration of potentially harmful vapors in the air over a defined sample period. To use, the tube is connected to a small vacuum pump that draws air through the tube at a precise flow rate over a specific period of time. XAD sorbent tubes are typically made of glass and contain various types of solid adsorbent material (sorbents). Air sampling methodology for the use of XAD sorbent tubes on this project can be found in the "Compendium of Methods for the Determination of Toxic Organic Compounds in Ambient Air Second Edition, Compendium Method TO-17" (EPA/625/R-96/010b).

The standard operating procedure for this method's sample collection is summarized as follows:

- Record the current sampling location on the appropriate field forms (include map ID and/or GPS coordinates).
- Calibrate each personal sampling pump with a representative sampler in line, record flow rate.
- Break ends of sampler immediately prior to sampling. Attach sampler to pump with flexible tubing.
- Sample at an accurately known rate for a specific duration.
- Cap the sorbent tube and pack securely for shipment with bagged refrigerant. Do not shut down the pump at this time.
- Each tube should go in a labeled Ziploc bag, noting sample ID and parameter, date and time. Labels should be prepared in such a way that they will not be ruined by water or refrigerant. Samples may be stored at 4C until they are shipped.
- Record the sampling information onto the Chain of Custody form.
- Using the same setup as for initial calibration, measure the flow rate again with the same dedicated sorbent tube specific to the pump used for that method. Record the final flow rate onto the Air Sampling Worksheet.

A.7 Quality Objectives and Criteria for Measurement Data

The data quality objective of this air sampling and analysis plan is to utilize TD and XAD sorbent tubes to perform air sampling around the AltEn facility wetcake holding areas and lagoon to potentially identify the source of existing odors being emitted. The results will provide a follow-up to the past EPA September 2021 air sampling event.

Representativeness will be addressed by collecting the samples as described in this document.

Comparability will be addressed by collecting, analyzing, and reporting the data as described in this document.

Air monitoring will consist of collection of air toxics data utilizing TD and XAD sorbent tube technology as described in the "Compendium of Methods for the Determination of Toxic Organic Compounds in Ambient Air Second Edition Compendium Method TO-17" (EPA/625/R-96/010b).

A.8 Special Training Requirements / Certification

Field sampling personnel will undergo some basic training on collecting air samples with TD and XAD sorbent tubes if they have not already done so as well as follow guidance found in R7 EPA SOP 1710.01A "Regional Field Personnel and Training."

All employees with project responsibility must read and understand the manufacturer's equipment documents, project quality assurance documents, and SOPs.

A.9 Documentation and Records

The Project Officer will have overall responsibility for assuring the most current approved QAPPs are being utilized through ongoing review of Region 7's QA process.

B.1 Sampling Process Design

Air samples are to be collected utilizing TD tubes and XAD sorbent tubes. Analysis will be performed by EPA ORD and done in accordance with the "Compendium of Methods for the Determination of Toxic Organic Compounds in Ambient Air Second Edition" (Compendium Method TO-17).

EPA Region 7 personnel will work directly with NDEE field personnel in selecting locations and taking air samples in and around the AltEn Ethanol facility at Mead, NE, focusing on the wetcake and lagoon holding areas as well as potential background sites. Sampling will occur over a two-day period and be conducted using pumped air (200 ml/minute) for 30 minutes in duration (as per ORD guidance) through 9 TD and 9 XAD sorbent tubes each day. A total of one duplicate, one field blank and one trip blank (see Section B.5.2) will be taken each day for a total of 24 samples for each respective tube sampling format for an overall total of 48 TD/Sorbent tube air samples.

The results will be reviewed for the identification of previously unidentified odors/contaminants and considered in deciding the need of future air toxics sampling events.

B.2 Sampling Methods Requirements

The methods for the Thermal Deposition Tube/ and XAD Sorbent Tube sampling was previously described in Section A.6.1, which outlines the air sampling methodology and performance requirements that will be implemented to ensure data is collected accurately. Specific guidance and further information can be found in the "Compendium of Methods for the Determination of Toxic Organic Compounds in Ambient Air Second Edition, Compendium Method TO-17" (EPA/625/R-96/010b).

B.3. Sampling Handling and Custody Requirements

Sample custody procedures are a vital aspect of any environmental sampling event. Each sample or field measurement must be properly documented to facilitate timely, correct, and complete analysis. The sample chain-of-custody procedure provides the means to identify, track, and monitor each sample from the point of collection through final data reporting.

A chain-of-custody record will be completed for each sample shipment. Standard laboratory chain-of-custody records will be used (Attachment A). After completion of the chain-of custody record, the original will then be enclosed in a sealable plastic bag and secured to the inside of the shipping container lid. A copy of the chain-of-custody record will be maintained in the project files. If more than one shipping container is used for a day's shipment, a separate chain of-custody record will be completed for each shipping container. The original chain-of-custody record received from the laboratory will be maintained in the final project file.

Shipping containers will be secured, and custody seals will be placed across the container openings. As long as the chain-of-custody record is sealed inside the shipping container and the custody seals remain intact, commercial carriers will not be required to sign the chain-of-custody record. Sample custody will be performed in accordance with R7 SOP 2420.05E "Identification, Documentation, and Tracking of Samples." Field work sheets will be used to track sample collection at the site. Original copies of field sheets will be maintained in the project file.

The sampling tubes are to be shipped in a cooler with ice packs. Further, the boxes of tubes will be placed in sealed plastic ziplok bags to keep them from getting wet. The air sampling tubes will be shipped to the ORD laboratory, where analysis is to take place within 24 hours of the last sample being completed and in accordance with R7 SOP 2420.07J, "STC Procedures for Sample Shipping to External Contract Laboratories."

B.4 Analytical Methods Required

Analytical methodology and procedures can be found in ORD air sampling tube analysis guidance the "Compendium of Methods for the Determination of Toxic Organic Compounds in Ambient Air Second Edition, Compendium Method TO-17" (EPA/625/R-96/010b).

B.5 Quality Control Requirements

Internal QC checks for sampling and sample analysis activities will be conducted in accordance with ORD guidance found in the "Compendium of Methods for the Determination of Toxic Organic Compounds in Ambient Air Second Edition, Compendium Method TO-17" (EPA/625/R-96/010b).

Internal QC procedures for the analytical laboratory supplying data are required and executed according to the ORD laboratory's SOPs and the "Compendium of Methods for the Determination of Toxic Organic Compounds in Ambient Air Second Edition, Compendium Method TO-17" (EPA/625/R-96/010b).

B.5.1 Duplicates

Duplicate sampling will be done at a frequency of no less than 10%, These samples will be prepared, handled, and analyzed in the same manner as regular samples. These samples will be identified within the data package as field duplicates. Upon data receipt, the field duplicate data will be reviewed for relative percent difference (RPD). Large relative percentage differences (RPDs) will be noted, and professional judgment will be used to qualify data when the RPD is > 50%.

B.5.2 Blanks

Field, trip, and laboratory blanks will be prepared and analyzed throughout the study to ensure sample integrity. Field and trip blanks will comprise 10% of the samples collected for each metric. Blanks will be representative of the media used to collect samples in the field.

Field Blanks

Field blanks are the same as laboratory blanks except that they are transported to and from the monitoring site. They are uncapped and immediately resealed at the monitoring site, but do not actually have air pumped through them. One field blank tube will be taken for every ten tubes used for this monitoring event with two field blanks collected for each type of sampling tube utilized.

Trip Blanks

Trip blanks will comprise 10% of the samples collected daily to document sample integrity associated with the shipment, collection, and storage of air toxics sampling. The trip blanks will be treated the same as samples, except they will not be removed from the transport container during sample deployment. The trip blanks will be stored with the samples following deployment to measure potential artifacts introduced during storage in the field and shipping for analysis.

Laboratory Blanks

The ORD laboratory will follow established guidance (the "Compendium of Methods for the Determination of Toxic Organic Compounds in Ambient Air Second Edition, Compendium Method TO-17" (EPA/625/R-96/010b) and internal QA to analyze a suitable number of blanks in accordance with guidance.

B.6 Instrument and Equipment Maintenance Requirements

The field equipment and analytical instrumentation testing, inspection, and maintenance will be performed in accordance with the above referenced analytical and sample collection SOPs along with manufacturer's recommendations.

B.7 Instrument / Equipment Calibration and Frequency

Field equipment and analytical instrument calibrations will be performed in accordance with the appropriate referenced analytical or sample collection guidance ("Compendium of Methods for the Determination of Toxic Organic Compounds in Ambient Air Second Edition, Compendium Method TO-17" (EPA/625/R-96/010b)) and manufacturer's recommendations.

Calibration activities utilizing the GilAir Plus (Sensodyne)Air Sampling Pump will be done according to information found in the GilAir Plus (Sensodyne) Air Sampling Pump Operation Manual.

B.8 Acceptance Requirements for Supplies and Consumables

No special requirements are needed.

B.9 Data Acquisition Requirements

There shall be no acquired data associated with this project.

B.10 Data Management

The analytical laboratory (ORD) will record sample analysis results in an electronic format.

C.1 Assessment and Response Actions

The EPA QA manager or their designee may conduct an audit of the field activities for this project if requested by the EPA project manager. The EPA QA manager will have the authority to issue a stop work order upon finding a significant condition that would adversely affect the quality and usability of the data. The EPA project manager will have the responsibility for initiating and

implementing response actions associated with findings identified during the on-site audit. Once the response actions have been implemented, the EPA QA manger will perform a follow-up audit to verify and document that the response actions were implemented effectively.

Assessments and response concerning the analytical aspect of the project are addressed in the appropriate EPA ORD SOPs and the "Determination of Toxic Organic Compounds in Ambient Air Second Edition, Compendium Method TO-17" (EPA/625/R-96/010b).

C.2 Reports to Management

The final report shall incorporate the results from the EPA air toxics sampling event at Mead, NE and shall be distributed in accordance with section A3. The final report will contain environmental sampling results. The sampling analysis report will not draw conclusions regarding potential human health impacts resulting from any analyte concentrations.

D.1 Data Review, Validation and Verification Requirements

The data will be peer reviewed by a qualified analyst and the ORD laboratory Section Manager.

The EPA project manager will be responsible for overall validation and final approval of the data in accordance with project purpose and use of the data in accordance with EPA Region 7 SOP 1720.03A "Regional Field Sample & Data Management."

D.2 Validation and Verification Methods

The data will be validated in accordance with EPA Region 7 SOP 2430.02I "Review of Data Deliverables Packages from CLP Contract Laboratories (Format, Procedures, and Content)" and the "Compendium of Methods for the Determination of Toxic Organic Compounds in Ambient Air Second Edition, Compendium Method TO-17" (EPA/625/R-96/010b).

D.3 Reconciliation with User Requirements

Once the data results are compiled, the EPA project manager will review the field duplicates to determine if they fall within the acceptance limits as defined in this QAPP. Completeness will also be evaluated to determine if the completeness goal for this project has been met. If data quality indicators do not meet the project's requirements as outlined in this QAPP (including the accuracy for lab spikes), the data may be discarded, and resampling may occur. The EPA project manager will evaluate the cause of the failure (if possible) and make the decision to discard the data and re-sample. If the failure is tied to the analysis, calibration and maintenance techniques will be reassessed as identified by the appropriate lab personnel. If the failure is associated with the sample collection and resampling is needed, the sampling personnel will be retrained.

E. References

Compendium of Methods for the Determination of Toxic Organic Compounds in Ambient Air Second Edition, Compendium Method TO-17 (EPA/625/R-96/010b).

Gilian GilAir (Sensodyne) Plus Air Sampling Pump Operation Manual

ATTACHMENT A

Standard Field Forms for Bridgeton Sanitary Landfill
Air Quality Assessment

CHAIN OF CUSTODY RECORD / ANALYTICAL REQUEST FORM

AltEn Air Toxics Sampling, Mead, NE

Client Name: USEPA Region7	Telephone No./ Fax No.: 913 551-5010	Date:	Page of
Project Manager: Leland Grooms.	REQUESTED TESTS / ANALYSES		
Address: 300 W Minnesota, Kansas City, KS 66101			
Project Name and Location: AltEn Air Toxics Sampling			
Sampled By:	Sampler Signature:		

LAB ID	SAMPLE ID NUMBER	Type	Date	Time	VOCS- EPA TO-17	-Other	Aldehydes- EPA TO-11A	Carboxylic Acids Tube GC-MS	HCL - NIOSH7903	Ammonia- OSHA ID-188	SO2 - OSHA ID200	HCN - NIOSH 6010	Amines- NIOSH 2010M	Fixed Gases - EPA 3C	PAHs / Dioxins EPA TO-13A / 9A	Mercury- NIOSH 6009	Odor Evaluation	Special Instructions / Conditions of Receipt

Requested Turnaround Time: As soon as possible			QC Requirements:		
Relinquished By:	Date:	Time:	Received By:	Date:	Time:
Relinquished By:	Date:	Time:	Received By:	Date:	Time:
Relinquished By:	Date:	Time:	Received By:	Date:	Time:

Air Toxics Monitoring Data Collection Sheet

AltEn Ethanol Facility, Mead, NE Air Toxics Sampling

Collected by_____ Date_____ Page _____of_____

Sample Number	Matrix	Latitude	Longitude	Initial Flow Reading	Final Flow Reading	Time Started	Time Ended	Total Time Sampled	Notes